



A Modified QuEChERS Method for the Determination of Organochlorine Pesticides in Serum

UCT Part Numbers:

ECQUUS1115CT - 800 mg MgSO_4 and 200 mg NaCl in 15 mL centrifuge tube

ECPURMPSMC - Quick QuEChERS - medium push through cartridge with 110mg MgSO_4 and 180mg PSA

GCLGN4MM-5 – Agilent system GC liner, 4mm splitless gooseneck, 4mm ID x 6.5mm OD x 78.5mm)

April 2015

Summary:

Organochlorine (OC) pesticides are hydrocarbons with multiple chlorine substitutions. They are primarily used as insecticides. OC pesticides do not break down easily as the chlorine-carbon bonds are very strong. As such they remain in the environment long after application and bio-accumulate in organisms long after exposure. They are toxic and some are known or suspected human carcinogens, such as 4,4'-DDT, 4,4'-DDD, and the lindanes. Many OC pesticides are banned or have restricted use. These chemicals are frequently found in human body tissue, blood, fat and breast milk. This application employs a modified QuEChERS extraction and a fast, push-through cartridge clean-up for the determination of OC pesticides in human serum.

1 mL of serum sample is mixed with 1 mL of reagent water and extracted with 2 mL of acetonitrile (MeCN). 800 mg magnesium sulfate (MgSO_4) and 200 mg sodium chloride (NaCl) are used to enhance the phase separation and the partition of the target analytes into the MeCN layer. After shaking and centrifugation, 1.5 mL of the supernatant is purified using a push through cartridge containing 110 mg MgSO_4 and 180 mg PSA. MgSO_4 absorbs any residual water in the extract, while PSA removes the matrix co-extractives, such as the lipids, resulting in a clean extract for instrumental analysis.

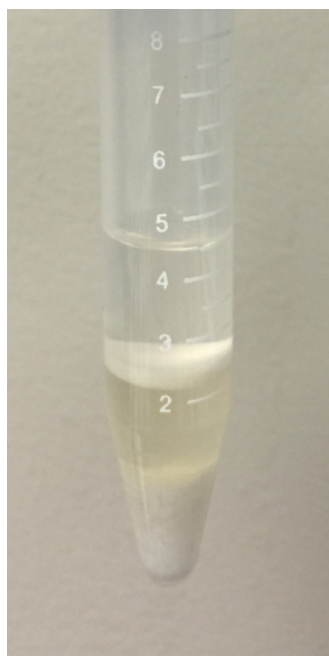
Procedure:

1. QuEChERS Extraction

- a) Add 2 mL MeCN containing triphenyl phosphate as internal standard (IS) to a 15-mL centrifuge tube pre-packed with 800 mg MgSO₄ and 200 mg NaCl (**ECQUUS1115CT**).
- b) Add 1 mL serum sample and 1 mL reagent water into the 15-mL centrifuge tube, and shake for 1 min manually or use a Spex 2010 Geno-Grinder at 1000 strokes/min.
- c) Centrifuge at 3000 g for 5 min.

2. Push Though Cartridge Cleanup

- a) Load 1.5 mL supernatant using a disposable plastic syringe and attach a push through cartridge (**ECPURMPSMC**) to the Luer lock tip of the syringe.
- b) Push the syringe plunger slowly, discard the first 3-5 drops (to remove any trapped airborne contaminants in the cartridge), then collect 0.5 mL extract into a 2-mL auto-sampler vial.
- c) The samples are ready for GC/MS (or GC-ECD) analysis.



Serum sample after QuEChERS extraction



Push through cartridge cleanup

GC/MS Method:

GC/MS	Agilent 6890N GC coupled to a 5975C MSD
Injection	1 μ L splitless injection at 250 °C
GC liner	4 mm splitless gooseneck (GCLGN4MM-5), packed with deactivated glass wool
GC column	Restek Rxi [®] -5sil MS 30m x 0.25mm, 0.25 μ m with 10m integrated guard column
Carrier gas	Ultra high purity Helium at a constant flow of 1.2 mL/min
Oven temp. program	Initial temperature at 50 °C, hold for 1 min; ramp at 10°C/min to 310 °C, hold for 3 min. Acquire data from 8 to 24 min.
Temperatures	Transfer line 280 °C Source 250 °C Quadrupole 150 °C

Compound Name	RT (min)	SIM Ions (25 ms)			R ²
TPP (IS)	22.643	326	325	77	NA
alpha lindane	15.911	181	219	109	0.9996
beta lindane	16.449	181	219	109	0.9995
gamma lindane	16.639	181	219	109	0.9998
delta lindane	17.195	181	219	109	0.9998
Heptachlor	18.185	100	272	237	0.9996
Aldrin	18.913	66	263	293	0.9998
Heptachlor epoxide	19.691	353	81	237	0.9995
trans Chlordane	20.155	373	237	272	0.9997
cis Chlordane	20.418	373	237	272	0.9998
Endosulfan I	20.418	237	195	339	0.9997
4,4'-DDE	20.812	246	318	176	1.0000
Dieldrin	20.951	79	263	277	0.9987
Endrin	21.357	263	81	281	0.9995
Endosulfan II	21.551	195	237	339	0.9995
4,4'-DDD	21.619	235	165	199	0.9992
Endosulfan sulfate	22.281	272	274	387	0.9984
4,4'-DDT	22.338	235	165	199	0.9973
Endrin ketone	23.186	317	67	319	0.9983
Methoxychlor	23.352	227	228	152	0.9970

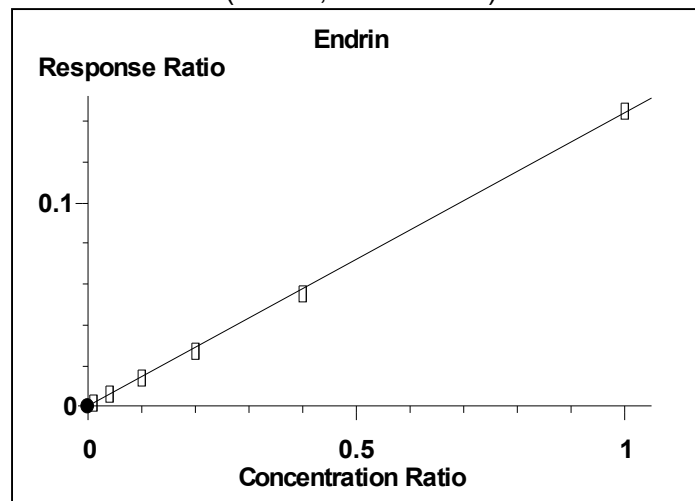
Results:

Recovery and RSD% of Spiked Serum Samples

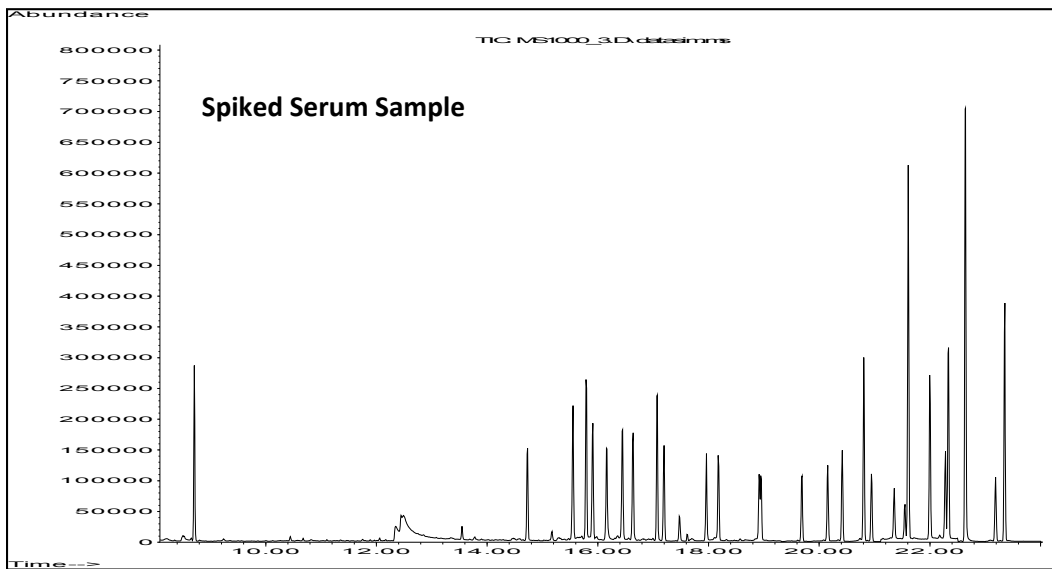
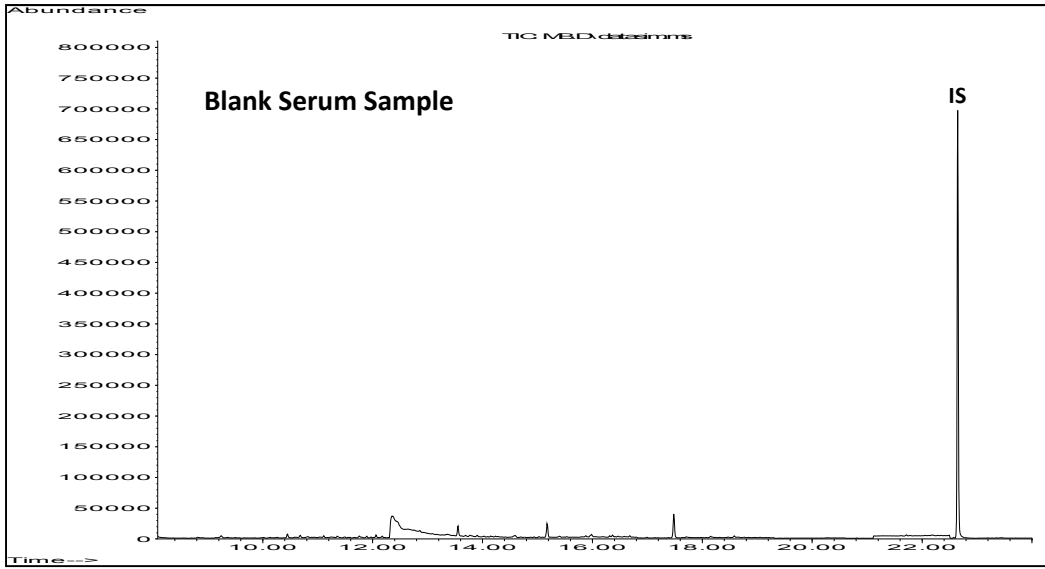
Compound Name	Spiked at 40 ng/mL		Spiked at 200 ng/mL		Spiked at 1000 ng/mL	
	Recovery%	RSD% (n=5)	Recovery%	RSD% (n=5)	Recovery%	RSD% (n=5)
alpha lindane	106.5	1.8	95.7	3.2	100.1	1.1
beta lindane	105.5	0.7	93.7	5.0	100.3	2.5
gamma lindane	110.2	1.9	97.6	3.3	101.2	1.3
delta lindane	101.4	1.5	94.5	3.5	101.0	1.0
Heptachlor	103.0	3.8	89.7	7.4	92.6	2.8
Aldrin	87.5	5.6	81.3	3.0	87.4	2.3
Heptachlor epoxide	98.7	2.2	90.2	3.2	99.6	2.5
trans Chlordane	90.1	3.2	84.3	3.7	92.7	2.6
cis Chlordane	90.8	3.4	83.4	3.4	92.5	1.4
Endosulfan I	96.9	3.1	88.3	4.0	94.4	1.3
4,4'-DDE	87.9	5.7	81.7	4.1	87.2	2.1
Dieldrin	101.5	0.7	89.2	3.4	95.1	4.2
Endrin	98.7	1.6	91.5	1.4	98.1	1.3
Endosulfan II	95.5	6.7	89.8	5.0	96.7	1.2
4,4'-DDD	77.8	2.6	79.2	3.1	94.6	1.1
Endosulfan sulfate	86.2	2.0	85.4	2.5	100.0	0.7
4,4'-DDT	61.6	4.7	65.2	3.1	87.0	5.1
Endrin ketone	91.7	2.2	89.4	3.8	99.5	1.4
Methoxychlor	69.8	2.3	74.1	3.0	96.0	0.7
Overall mean	92.7	2.9	86.5	3.6	95.6	1.9

Matrix Matched Calibration Curve

(Endrin, $R^2 = 0.9995$)



Chromatograms



5104-04-01